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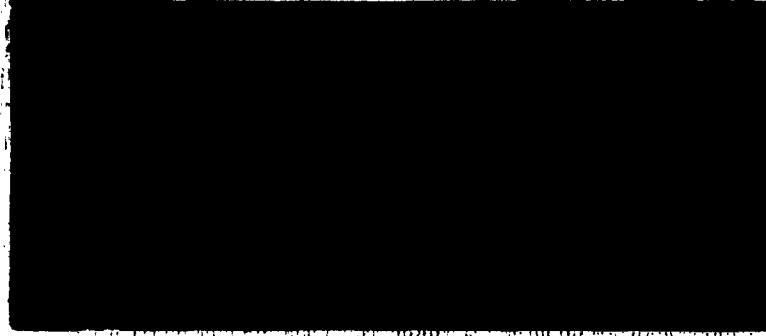
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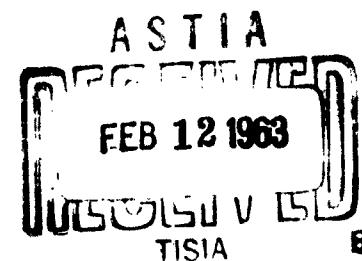
**EVALUATION OF MOLYBDENUM METAL
PRODUCED BY THE TIN REDUCTION
OF MOLYBDENUM DISULPHIDE PROCESS**

Contract N0w-62-0754-c

(Quarterly Report No. 3)

13 October 1962 - 13 January 1963

**Department of the Navy
Bureau of Naval Weapons
Washington 25, D. C.**



EVALUATION OF MOLYBDENUM METAL PRODUCED BY THE TIN REDUCTION OF MOLYBDENUM DISULPHIDE PROCESS

I. SYNOPSIS OF PREVIOUS REPORT PERIOD

The reduction facility was cleaned and repaired. Several trial runs were made to test the unit components.

An unusually high fixed carbon was found in Grade I Climax molybdenite. This carbon was successfully removed by roasting in wet hydrogen at 800°C. The roasting process also made a contained iron fraction amenable to further removal by hydrochloric acid leaching. A substantial quantity of molybdenum oxide soluble in acetone was found after acid leaching and was presumed to be "Molybdenum Blue" so the molybdenite was acetone leached and dried. Analyses on molybdenite processing are incomplete at this time and will be included in the next quarterly report.

II. SUMMARY OF PRESENT REPORT PERIOD

A total of 160 pounds of molybdenum was produced by the tin reduction process. One experimental molybdenum ingot Mo-1 was final melted and sectioned for testing. Tensile test and bend test specimens were made from as cast material as well as two blanks for swaging. After swaging one rod is severely fractured. The second rod is being tested for soundness.

A second experimental ingot was first melted and a molybdenum ingot produced from commercial molybdenum powder was first melted.

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Carbon in high concentrations in molybdenite was found hard to remove completely. Extensive processing of MoS₂ with wet hydrogen continues with particular attention focused on obtaining good random samples of processed material for analyses. This fixed carbon in molybdenite was not found to be a problem in previous work two years ago and thus has caused an unexpected delay in reduction while low carbon MoS₂ is being produced.

The "Molybdenum Blue" material isolated from previous molybdenite leaching with acetone was amorphous and thus not amenable to X-ray diffraction identification. Since only a small amount of material was isolated, further identification work was impossible.

III. PRESENT REPORT PERIOD

A total of 160 pounds of molybdenum metal powder was produced by the Nachtmann-Poole process during this period. One 5-inch diameter ingot weighing about 40 pounds as cast was produced from this powder and sectioned for testing and chemical analysis. Two swaging blanks were sawed for tensile testing from the longitudinal axis and two blanks were sawed from the horizontal axis of the ingot. These four samples are currently being machined for testing. Ten bend test specimens 1.4" X 1.2" X 0.100" were sawed longitudinal to the ingot axis and are being machined for testing. One hundred grams of the ingot were crushed to -20 mesh grain in a diamond mortar for chemical and spectrographic analysis. The impurity analysis of the raw

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material going into the ingot, the powder produced, and the melted ingot are as follows:

TABLE I
FINAL ANALYSIS OF CLIMAX GRADE I MOLYBDENITE, IN PPM

C	O
50	30

TABLE II
ANALYSIS OF A SPLIT SAMPLE OF TIN REDUCED MOLYBDENUM POWDER
PRODUCED FOR INGOT Mo-1, IN PPM*

C	O	W	Sn	Spec Imp Total
<20	150	2200	300	<50

*Oxygen not typical, sample exposed to air 3 hours prior to arc melting sample button.

TABLE III
ANALYSIS OF INGOT Mo-1 PRODUCED FROM NACHTMANN-POOLE
PROCESS MOLYBDENUM POWDER, IN PPM

C	O	Al	Co	Cr	Cu	Fe	Mg	Ni	Si	Ti	Sn	W
700	100	<50	<50	<50	<50	100	<50	<50	50	150	50	3100

The high carbon must have come from molybdenite feed material. There must have been a localized area of high carbon MoS_2 in this lot. The following carbon values in Table IV were obtained on samples of the MoS_2 run through the decarbonization retort the first time:

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TABLE IV

CARBON ANALYSES RUN ON EACH BATCH OF MoS₂ THROUGH
WET HYDROGEN RETORT PROCESS, IN PPM

	C
Batch 1	90
Batch 2	80
Batch 3	90
Batch 4	160
Batch 5	80
Batch 6	180

All of the molybdenite was run through the wet hydrogen process once again, acid leached, and a sample of the above combined lot taken. This sample was briquetted with tin and reduced to metal. The molybdenum powder thus produced analyzed 50 PPM carbon and 30 PPM oxygen as shown in Table I. This molybdenite was used in producing all of the 160 pounds of molybdenum powder made to date. Buttons were made during powder production as spot checks on purity and from visual analysis contained carbon and oxygen levels comparable to the above sample. Therefore, a localized high carbon area in the MoS₂ must have been present.

Test specimens from ingot Mo-1 cut easily, the hardness measuring R_b 79. A small arc cast sample was rolled successfully at 1000°C with no edge cracking noted at about 75% reduction. Two rods 1-11/32" diameter by 3" long were machined for

swaging and were swaged to about 5/8" diameter for testing. One rod was severely cracked over 3/4 of its length and the other rod is in process of dye check to determine whether it is sound throughout its length.

A total of 70 pounds of commercial molybdenum powder was compacted into 1" X 1" X 15" bars and sintered 24 hours at 1400°C - 1450°C following the identical procedure used for processing tin reduced molybdenum powder bars for melting. About half of these bars have been consumably arc melted one time into a 2-1/2" diameter crucible. Upon completion of the first melts, the metal will be consolidated into one 3-1/2" diameter ingot and remelted again into a 5-1/2" crucible. This sequence follows the pattern set up for producing ingots from Nachtmann-Poole process molybdenum.

A total of 50 pounds of tin reduced molybdenum powder for a second ingot has been consolidated into 2-1/2" diameter first melt ingots and was subsequently converted into a 3-1/2" ingot. A total of 50 pounds of tin reduced molybdenum powder has been compacted into 1" X 1" X 15" bars for melting and will be melted after commercial molybdenum powder melting has been completed.

The Climax Grade I disulfide received contains a high level of fixed carbon. A second 200 pound batch of molybdenite after acid leaching contained 3/4% carbon. After processing in wet hydrogen at a furnace temperature of 850°C for 4 hours, carbon was lowered to 1/2%. After a second wet hydrogen treat-

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ment with increased hydrogen flow (about a 10-fold increase in wet hydrogen flow) the carbon was lowered to about 0.3%. A larger reduction in carbon content was expected at the increased wet hydrogen flow rate but was not achieved because the cooling effect of the increased gas flow rate lowered the temperature of the MoS₂ bed during the run. Wet hydrogen will be passed through the MoS₂ again at an increased temperature after modification to insulate against heat losses. The molybdenite will be acid leached with hydrochloric acid and reduced with tin as before when all carbon is removed. The stainless steel processing retort oxidizes slowly during carbon removal leaving abraded oxide particles in the MoS₂. This material can be removed quite effectively by acid leaching in hydrochloric acid.

IV. FUTURE WORK

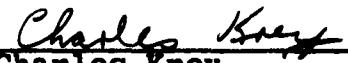
Reduction of MoS₂ has progressed very well. This phase of the process has proven to be trouble free and will continue when low carbon molybdenum disulfide has been produced.

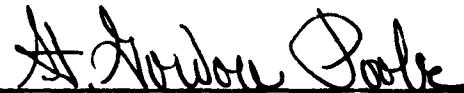
Melting and testing will progress according to the planned outline submitted in our previous report with the final report to be presented by April 13, 1963.

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Respectfully submitted,

OREGON METALLURGICAL CORPORATION


Charles Krey
Development Engineer


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